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# Unpolluted fractionation of wheat straw by steam explosion and ethanol extraction

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#### Abstract

An unpolluted process of wheat straw fractionation by steam explosion coupled with ethanol extraction was studied. The wheat straw was steam exploded for 4.5 min with moisture of 34.01%, a pressure of 1.5 MPa without acid or alkali. Hemicellulose sugars were recovered by water countercurrent extraction and decolored with chelating ion exchange resin D412. The gas chromatography (GC) and high-performance liquid chromatography (HPLC) analysis results indicated that there were organic acids in the hemicellulose sugars and the ratio of monosaccharides to oligosaccharides was 1:9 and the main component, xylose, was 85.9% in content. The total recovery rate of hemicellulose was 80%. Water washed materials were subsequently extracted with ethanol. The optimum extraction conditions in this work were 40% ethanol, fiber/liquor ratio 1:50 (w/v), severity  $\log(R) = 3.657$  (180 °C for 20 min), 0.1% NaOH. The lignin yield was 75% by acid precipitation and 85% ethanol solvent was recovered. The lignin was purified using Björkman method. Infrared spectrometry (IR) results indicated that the lignin belonged to GSH (guaiacyl (G) syringyl (S) and *p*-hydroxyphenyl (H)) lignin and its purity rate reached 85.3%. The cellulose recovery rate was 94% and the results of electron spectroscopy for chemical analysis (ESCA) and infrared spectrometry (IR) showed that hemicellulose and lignin content decreased after steam explosion and ethanol extraction. © 2006 Elsevier Ltd. All rights reserved.

Keywords: Fractionation; Steam explosion; Ethanol extraction

#### 1. Introduction

Efficient separation of constitutive biomass components constitutes one of the major obstacles to the efficient utilization of renewable resources. However, such separation is mandatory if sustainable derived plant (renewable) resources are to advance as feedstocks for chemicals and materials which are currently obtained from fossil carbon resources (Myerly et al., 1981; Bozell et al., 1995). The extraction of lignin and noncrystalline carbohydrates (hemicellulose or polyoses) from wood is commercially practiced by the pulp and paper industry. However, while the paper industry has managed to define conditions under which cellulose-rich pulp fibers could be isolated and purified efficiently, the process has failed to achieve efficient

fractionation performance by wasting (through underutilizing the noncellulosic biomass as process fuel) half of the plant resource (Ibrahim and Glasser, 1999). Bozell brought forward the conception of "clean fractionation" and its five criteria (selectivity, accessibility, recoverability, utility and economics) in 1995.

Most fractionation processes were based on the use of a physicochemical pretreatment that increased the reactivity of the lignocellulosic structure, followed by one or more separation and purification stages to isolate products. Hydrolytic treatment of lignocelluloses by saturated steam or hot water (autohydrolysis), or with addition of small amounts of acids or alkali (prehydrolysis), has been widely studied as a method to weaken the lignocellulosic structure and increase its chemical reactivity and enzyme accessibility. A variety of processes aimed at biomass fractionation have been proposed and investigated based on this approach (Knappert et al., 1980; Biermann et al., 1984;

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Heitz et al., 1991; Wallis and Wearne, 1992; Montané et al., 1993, 1998; Glasser and Wright, 1998). This approach has been referred to as steam explosion. However this approach does not separate lignin from the lignocellulosic materials (Excoffer et al., 1991).

Kleinert (1974) has studied the mechanism of ethanol pulping and it is reported to be a cleaner delignification method. Now organosolv pulping processes have been presented as proven alternatives to the Kraft process due to their economy, environmental amity and ability to produce substantial quantities of useful by-products (lignin, sugars, furfural and acetic acid) in addition to pulp (Laxen, 1987; Williamson, 1987; Dahlmann and Schroeter, 1990; Ni et al., 1997; Botello et al., 1999). Ethanol is still one of the most widely used organic solvents (Asiz and Sarkanen, 1989; Oliet et al., 2000, 2002).

Most studies focused on wood while fractionation of wheat straw has been paid less attention despite the magnitude of the annual production of straw (147 mt/year in China). Research on wheat straw fractionation has been centered on the pretreatment by dilute acid hydrolysis (Knappert et al., 1980; McMillan, 1994) or autohydrolysis (Garrote et al., 1999). Complete fractionation sequences combined steam pretreatment at low temperature with delignification by alkali and ethanol (Papatheophanus et al., 1995), and at high temperature followed by hemicellulose extraction and alkali delignification.

Previous work (Chen et al., 1999) has given the optimum condition of steam explosion to pretreat wheat straw. The

aim of this work was fractionation of wheat straw by a process sequence based on our previous work (Chen et al., 1999), involving autohydrolysis and lignin extraction by ethanol at alkaline conditions followed by hemicellulose and lignin purification.

#### 2. Methods

#### 2.1. Materials

Chipped wheat straw (2.5 cm in length) was kindly provided by Tong County Beijing Papermaking Plant, Beijing. It contains 35.1% cellulose, 5.3% Klason lignin, 27.1% hemicellulose and 6.04% ash. All standard chemicals, including xylose and xylan, were purchased from Sigma Chemical Company (Beijing, PR China).

#### 2.2. Fractionation process

A flow chart of the fractionation procedure was shown in Fig. 1. The chipped wheat straw was initially steam exploded, and subsequently water washed to fractionate the hemicellulose. The remains were ethanol extracted to separate the cellulose and lignin.

#### 2.2.1. Steam explosion

Steam explosion pretreatment was performed in an 8.8 L reaction vessel designed by the Chinese Academy of Forestry according to the patent of Iotech (Delong,

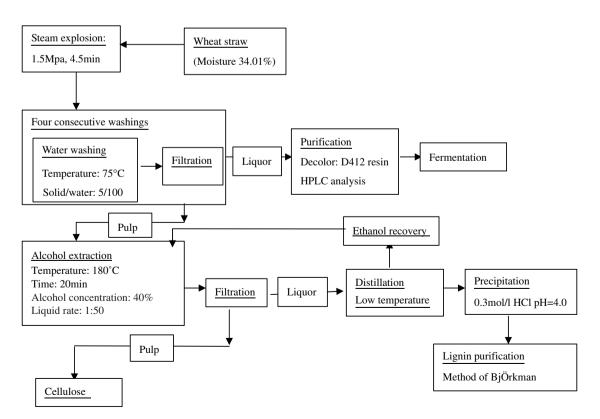


Fig. 1. Flow diagram of the fractionation process and operational conditions used in each stage.

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